Thermally activated spectroscopy of optical absorption in $Bi_{12}SiO_{20}$ crystals

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Abstract. We suggest applying a method of thermally activated spectroscopy to the problem of impurity optical absorption. The method consists in measuring the temperature dependence of optical absorption in a wide-gap semiconductor crystal and analyzing the temperature derivative of this dependence. The above technique allows for determining the energies of thermal and optical activations and the strength of electron–phonon interaction for the impurity centres.

1. Introduction

Methods of thermo-stimulated spectroscopy play an important role in the studies of intrinsic and impurity defects in semiconductor and dielectric crystals. Up to date, thermo-activated methods have been developed for the luminescent, current and capacitive spectroscopies [1, 2]. They yield in different quantitative characteristics of the trapping centres of charge carriers, which form the energy spectrum of local levels in the forbidden gap of crystals. The same refers to the parameters of electrically active defects, such as dipoles and quasi-dipoles. In addition, the methods mentioned above make it possible to find the characteristics of many electrophysical phenomena arising in thermodynamically non-equilibrium states, including those occurring in metal–dielectric–metal or metal–dielectric–semiconductor structures.

High-resistance semiconductors and dielectrics are important objects for many applications, for which the methods of thermally activated spectroscopy are widely applied. They are employed in semiconductor and functional electronics, including photonics and optoelectronics. A controlled presence of defects (e.g., intrinsic or impurity centres) makes it possible to modify the properties of these materials. At the same time, there still remain a number of questions concerned with both electrical and optical activities of these defects, which are not answered by the common methods of thermo-activated spectroscopy mentioned above. In particular, these techniques cannot ascertain the ratio of the energies of thermal and optical activations of defects, the strength of electron–phonon interaction and the nature of local energy levels available in the forbidden gap.

In this study we suggest supplementing the information provided by the thermo-activated spectroscopy with experimental study and theoretical analysis of the temperature dependences of optical absorption in wide-gap semiconductor and dielectric crystals. Our idea is based on the general principles that underlie the methods of thermo-activated spectroscopy. One of them is that a crystal is first brought into a thermodynamically non-equilibrium state and then a process of its

thermally stimulated relaxation is observed. Unlike the approaches known earlier, the relaxation characteristic to be recorded by us is not the electrical conductivity (bias current) or the luminescence. Instead, we suggest analyzing the impurity-induced optical absorption.

Specific subjects of our experimental studies are photorefractive crystals of silico-sillenite, $Bi_{12}SiO_{20}$ (BSO), which belong to a sillenite $Bi_{12}MO_{20}$ family (with M denoting Si, Ge and Ti). Since these crystals manifest a unique combination of practically useful properties, they have been comprehensively investigated. In the recent decades, the sillenites have become a promising active material used, e.g., in many devices for recording and processing optical information, and in holographic interferometry [3, 4]. The technical characteristics of $Bi_{12}MO_{20}$ have been determined from the photoelectric processes. In particular, the effect of Al, Ga, Cr, Mn, Cu and Mo impurities on the optical absorption, photoconductivity and the photochromic effect (PCE) in the $Bi_{12}MO_{20}$ crystals has been studied. It is known that Al and Ga ions weaken the PCE, whereas Cr and Mn ions enhance it [5, 6].

In the present work we study the spectral and temperature dependences of optical absorption in the pure (undoped) BSO crystals and the crystals doped with Al, Ga and Cr. Hereafter they are referred to as BSO, BSO:Al, BSO:Ga and BSO:Cr, respectively.

2. Experimental

The BSO, BSO:Al, BSO:Ga and BSO:Cr crystals were grown by the Czochralski technique. The amount of doping impurities was equal to 0.1 mass % (Al), 0.02 mass % (Ga) and 0.02 mass % (Cr). The samples were prepared in the shape of optically polished plates with the thickness $d = 0.1 \div 5.0$ mm.

At the first stage of our experiments, the spectra of optical transmission for the stationary $\left(t_0 \left(h\nu \right) \right)$ and thermodynamically non-equilibrium $\left(t^* \left(h\nu \right) \right)$ states of the samples were measured in the vicinity of the temperature point 80 K, using Specord M40 and Cary 5E spectrophotometers. After that, the stationary $\left(\alpha_0 \left(h\nu \right) \right)$ and photo-induced $\left(\alpha^* \left(h\nu \right) \right)$ absorption spectra were calculated, where a known optical-reflection spectrum for BSO was taken into account. The spectral range for the energy of light quanta was $h\nu = 0.5–3.4$ eV. A transition into non-equilibrium photo-induced state was achieved by exciting the samples with ultraviolet light. The PCE spectra were calculated as $\alpha^{PE} \left(h\nu \right) = \alpha^* \left(h\nu \right) - \alpha_0 \left(h\nu \right)$.

At the second stage, the temperature dependences of photo-induced optical absorption $\alpha^{PE} \left(T, h\nu_{i\max} \right)$ were recorded at every ith spectral position $\alpha^{PE} \left(h\nu \right)$ where a maximum (i.e., absorption peak) takes place. The temperature was changed in the two intervals $\Delta T_1 = 80–280$ K and $\Delta T_2 = 300–700$ K according to the linear law $T = T_0 + \beta t$ (with $T_0 = 80$ K). This was done in the heating run, with the rate $\beta = 0.16$ K×s^{-1}. The 'reverse' dependences $\alpha^{PE} \left(T, h\nu_{i\max} \right)$ were recorded during the cooling run.

3. Experimental results and their discussion

As seen from Fig. 1, the PCE spectra $\alpha^{PE} \left(h\nu \right)$ for all the crystals reveal a significant effect of impurities on the intensity and spectral structure of the photo-induced absorption. This refers to both non-transition metals (Al and Ga) and transition metals of iron group (Cr). In general, the PCE spectra for the BSO, BSO:Al and BSO:Ga crystals are consistent with those reported earlier [5], while the spectra of BSO:Cr reveal some new structural details [6]. To study the temperature dependences of the PCE relaxation $\alpha^{PE} \left(T, h\nu_{i\max} \right)$, we have taken the following facts into

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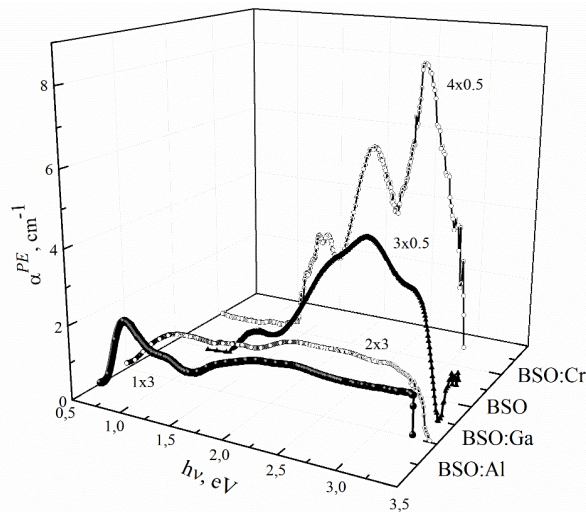
Fig. 1. PCE spectra for the BSO:Al (1), BSO:Ga (2), BSO (3) and BSO:Cr (4) crystals at T = 80 K.

account. The PCE in the BSO:Al and BSO:Ga crystals is weak, in contrast to the effect occurring in the undoped BSO and BSO:Cr crystals. It reveals the characteristic bands of photo-induced absorption in the near-infrared range (in particular, those described by the peaks $hv_{1\,max}$ = 0.78 eV for BSO:Al and $hv_{2\,max}$ = 0.81 eV for BSO:Ga – see Fig. 1). However, the PCE is not observed at $T > 300$ K.

As a consequence, the temperature dependences of the PCE relaxation $\alpha^{PE}\left(T, hv_{1,2\,max}\right)$ for the BSO:Al and BSO:Ga crystals have been studied in the low-temperature range ΔT_1 in the region of the bands mentioned above. The PCE in undoped BSO is pronounced enough (Fig. 1) and occurs in the both intervals ΔT_1 and ΔT_2. Then the dependences $\alpha^{PE}\left(T, hv_{3,4\,max}\right)$ for this crystal have been studied in these intervals at the photon energies close to the characteristic bands with the peaks $hv_{3\,max}$ = 1.5 eV and $hv_{4\,max}$ = 1.75 eV.

Furthermore, we take into account that the Al^{3+} and Ga^{3+} ions, which replace Si^{4+} in the BSO lattice, are acceptors. In addition, both acceptors and donors arise in the BSO crystals due to the presence of their intrinsic defects. Hence, the BSO, BSO:Al and BSO:Ga crystals in their thermodynamically equilibrium state occurring at low temperatures represent compensated wide-gap semiconductors, with ionized donors and accentors. The transition into non-equilibrium state, which is photo-activated by ultraviolet quanta with the energies greater than the forbidden gap, leads to the PCE and neutralization of the donors and acceptors. The latter create local energy levels inside the forbidden gap, which can take part in thermally stimulated exchange by electrons with either conduction or valence band. We assume that the condition $hv_{i\,max} = E_{ai}^{O}$ holds true, where E_{ai}^{O} denotes the optical activation energy of the defects responsible for the selected bands in the $\alpha^{PE}\left(hv\right)$ spectra. As a result, to study the temperature dependences of the intensity of photo-induced absorption bands in the PCE spectra, we select the bands for which the optical activation energy E_{ai}^{O} (i = 1, 2, 3 and 4) of the corresponding defects does not exceed half of the forbidden gap for the BSO crystals ($\Delta E_g \approx$ 3.4 eV at 80 K).

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The bands of the stationary absorption are located inside the following four intervals: A (0.5–1.4 eV), B (1.4–2.0) eV, C (2.00–2.75) eV and D (2.75–3.50) eV. Due to the PCE, the intensity of weak narrow A-bands with $h\nu_{max} = 0.800$, 1.195 and 1.239 eV decreases, though it increases for the band $h\nu_{max} = 1.072$ eV, as well as in the regions of the B, C, and D bands (see Fig. 1). The spectral positions and the intensity ratios for these bands allow us to associate them with the electronic $d-d$ transitions of $3d^3$ configuration in Cr^{3+}. Here the $^4A_2$, $^4T_2$, $a\,^4T_1$ and $b\,^4T_1$ quartet terms are relevant. The orbital singlet $^4A_2$ is the main term for any crystal field strengths Dq. The transitions from this level to the $^4T_2$, $a\,^4T_1$ and $b\,^4T_1$ triplets result respectively in the wide bands termed as U, Y and V. Taking U as the main band from the B interval, we obtain $Dq = 1.67$ eV (or $Dq = 1.73$ eV if the electron–phonon interaction is accounted for). This is an acceptable value for the transitions in the Cr^{3+} ions, which supposedly replace Bi^{3+} and lie in the oxygen octahedrons with large 'ligand–impurity ion' distances.

However, these bands are superimposed on the PCE spectrum of undoped BSO, except for the П-shaped band located at $h\nu_{5\,max} = 1.93$ eV (see Fig. 1). The temperature dependences of intensities of the intra-centre absorption bands and the 'impurity level – band' absorption are different. This is why we assume that these dependences can indicate the contributions of the intrinsic defects in BSO into the PCE occurring in the BSO:Cr crystals.

The dependences $\alpha^{PE}(T, h\nu_{i\,max})$ (with $i = 1$, 2 3, 4 and 5) have a stepwise decreasing shape (see Fig. 2). The numbers of steps, their temperature positions and widths depend on the type of crystal, provided that the heating rate and the temperature range under study are the same. We assume that the stepwise absorption decrease is due to depleting of the neutral donor levels (or filling of the acceptor levels) and changes in the intensities of intra-centre transitions in the BSO:Cr crystals.

Let us take into account that the temperature dependence of optical absorption occurring in the case of thermal emptying of neutral donors with the concentration N_d can be written as

$$\alpha^{PE}(T, h\nu_{i\,max}) = S_d^{OP}(T) N_d(T), \tag{1}$$

where S_d^{OP} is the cross section of photo-ionization of a donor level. A similar relation holds true for the case of acceptor filling. It is known that, during electron–phonon interaction, the S_d^{OP}
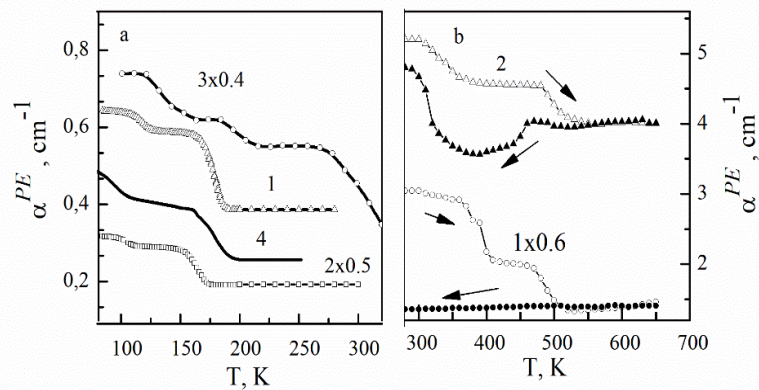
Fig. 2. Experimental (curves 1, 2 and 3 in panel a, and curves 1 and 2 in panel b) and calculated (curve 4 in panel a) $\alpha^{PE}(T)$ dependences for the BSO:Al (panel a, curves 2 and 4), BSO:Ga (panel a, curve 1), BSO (panel a, curve 3, and panel b, curve 1) and BSO:Cr (panel b, curve 2) crystals. Curve 4 is shifted upwards along the α^{PE} axis by 0.2 cm^{-1}. Arrows in Fig. 2b indicate the change of direction of temperature (cooling–heating).

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value decreases monotonically with increasing temperature. The effect can be approximately described by the relation [7]

$$S_d^{OP}(T) = S_d^* \left(\frac{T}{T_0} \right)^{-0.5},$$

(2)

with $S_d^* \approx$ const.

To find the N_d parameter, we consider that the effective cross section for thermal capture of electrons into the conduction band equals to

$$\sigma_s^T = \sigma_{s0}^T \left(\frac{T}{T_0} \right)^{-0.5} \exp\left(\frac{-E^T}{kT} \right),$$

(3)

where $T_0 = 70$ K, $\sigma_{s0}^T \approx const$ and E^T is the activation energy of donor levels with respect to the bottom of the conduction band [7]. The flux e_n of thermally activated electrons from the donor levels into the conduction band is determined by the well-known expression:

$$e_n = W_n^T \sigma_s^T \upsilon_T N_c,$$

(4)

where $W_n^T = \exp\left(\frac{-E^T}{kT} \right)$ is the probability of thermal activation of donors, $N_c = N_{c0} \left(\frac{T}{T_0} \right)^{1.5}$ the density of states in the conduction band, and $\upsilon_T = \upsilon_{T0} \left(\frac{T}{T_0} \right)^{0.5}$ the electron velocity. When examining Eq. (3), one can see that the pre-exponential factor in Eq. (4) is proportional to $\left(\frac{T}{T_0} \right)^{1.5}$ and so can be neglected because it is characterized by weaker temperature dependence than that governed by the exponential factor.

There is a simple relation $N_d^i = N_{d0} - N_d$ between the concentrations of neutral and ionized donors, with N_{d0} being the initial concentration of the neutral donors at $T = T_0$. The kinetic equation characterizing a decrease in N_d reads as

$$\frac{dN_d}{dt} = -N_d e_n.$$

(5)

Basing on the relation $T = T_0 + \beta t$, one gets the formula

$$\frac{dN_d}{dT} = -\beta^{-1} N_d e_n.$$

(6)

From Eq. (6) one has

$$N_d = N_{d0} \exp\left[-\int_{T_0}^T \beta^{-1} \tau_0 \exp\left(-\frac{E_a^T}{kT'} \right) dT' \right],$$

(7)

where $\tau_0 = \sigma_{s0}^T N_{c0} \upsilon_{T0}$. Here $E_\sigma^T + E^T = E_a^T$ and N_{d0} are the effective thermal activation energy and the initial concentration of neutral donors, respectively.

Issuing from Eqs. (1), (2) and (3), we obtain

$$\alpha^{PE} = S_d^* N_{c0} \left(\frac{T}{T_0} \right)^{-0.5} N_d(T).$$

(8)

The following formula has been obtained for the two-step reduction of the photo-induced absorption:

$$\alpha^{PE}(T) = S_{d1}^*\left(\frac{T}{T_0}\right)^{-0.5} N_{d1}(T) + S_{d2}^*\left(\frac{T}{T_0}\right)^{-0.5} N_{d2}(T) + \alpha_0 , \qquad (9)$$

where α_0 is the residual absorption, N_{d1} and N_{d2} are the concentrations of donors with the effective activation energies E_{a1}^T and E_{a2}^T, and S_{d1}^* and S_{d2}^* are the constants of photo-ionization cross sections.

The electron concentration n in the conduction band increases with increasing concentration of the ionized donors as follow:

$$\frac{dN_d^i}{dT} = \beta^{-1} N_d e_n = An , \qquad (10)$$

where A denotes a proportionality coefficient. Finally, we arrive at the formula

$$An = \beta^{-1}\tau_0 N_{d0} \exp\left[-\frac{E_a^T}{kT} - \int_{T_0}^{T} \beta^{-1}\tau_0 \exp\left(-\frac{E_a^T}{kT'}\right)dT'\right]. \qquad (11)$$

Eq. (11) is similar to the formula known for the thermally stimulated current $j(T) = A_1 n(T)$ (with $A_1 = q\mu E$, q and μ being respectively the charge and the mobility of electrons, and E implying the electric field strength. The A_1 parameter is only weakly dependent on temperature. In this case we have

$$j(T) = A_1 \exp\left[-\frac{E_a^T}{kT} - \int_{T_0}^{T} \frac{A_2}{\beta} \exp\left(-\frac{E_a^T}{kT'}\right)dT'\right], \qquad (12)$$

where $A_2 \approx const$.

Neglecting the dependence $S_d^{OP}(T)$, one can assume that $F(T) = \dfrac{\partial\left(\alpha^{PE}(T, h\nu_{i\,max})\right)}{\partial T} \approx An = \dfrac{dN_d^i}{dT}$. The dependences $F(T)$ found by us as the temperature derivatives of experimental dependences $\alpha^{PE}(T, h\nu_{i\,max})$ are presented in Fig. 3 (see curves 1, 2 and 3 in panel a, as well as curves 1 and 2 in panel b). The general appearance of the $F(T)$ curves and the positions T_{max} of their peaks correlate well with the shape and peak positions detected for the thermally stimulated current $j(T)$ in the undoped and doped BSO crystals (see Table 1).
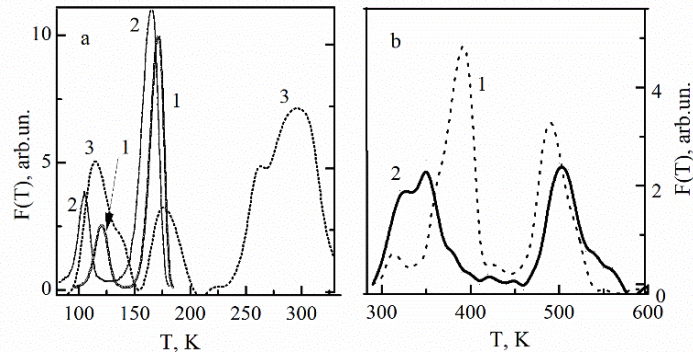
Fig. 3. Derivatives $F(T)$ of the temperature dependences of photo-induced optical absorption $\alpha^{PE}(T)$ for the BSO:Al (curve 1 in panel a), BSO:Ga (curve 2 in panel a), BSO (curve 3 in panel a and curve 1 in panel b) and BSO:Cr (curve 2 in panel b) crystals.

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Table 1. Parameters of local centres obtained with the method of thermally activated spectroscopy of optical absorption and the thermally activated current spectroscopy [1]

Crystal	Data of thermally activated spectroscopy of optical absorption				Data of thermally activated current spectroscopy		
	T_{max} , K	E_a^T , eV	E_a^O , eV	E_a^O / E_a^T	T_{max} , K	E_a^T , eV	Reference
BSO	112	0.16*	1.50	9.4	95–100	0.20*	[8]
	176	0.27*	"	5.6	170–180	0.41*	[8–10]
	219	0.40*	"	3.8	210–220	0.43, 0.33*	[8–11]
	264	0.36*	"	4.2	240–260, 264	0.50, 0.49*	[8–11]
	300–313	0.56*	1.75	3.1	300–320	0.68*	[8–11]
	490	0.85*	"	2.1	480–490	0.85*	[11]
	520	0.90**	"	2.0	525	1.00*	[11]
BSO:Al	103	0.15*	0.78	5.2	100	0.30*	[12]
	164	0.35*	"	2.2	155	0.30–0.70	[12, 13]
BSO:Ga	119	0.13*	0.81	6.2	120	0.24*	[14]
	166	0.25*	"	3.2	170	0.29*	[14]
BSO:Cr	325	0.55*	1.93	3.5	–	–	–
	350	0.61*	"	3.2	363	0.51**	[15]
	509	0.99*	"	2.0	483	0.90**	[15]

[1] Figures marked as * and ** correspond to the E_a^T values obtained by analyzing the $F(T)$ peaks respectively with the initial-slope and Urbach methods ($E_a^T = AkT_{max}$, where $A = 19$ for BSO [11]).

Since Eqs. (11) and (12) for $F(T)$ and $j(T)$ are similar, we use the standard methods of thermally activated current spectroscopy in analyzing the $F(T)$ peaks [1, 2]. In particular, an 'initial-slope method' is the most accurate. It allows one to find the thermal activation energy E_a^T of the donor (acceptor) levels issuing from the $F(T)$ dependences presented in the Arrhenius coordinates [1, 2]. The E_a^T values calculated in this manner are consistent with the data reported earlier using the analysis of peaks in the $j(T)$ curves and employing the common methods of thermally activated current spectroscopy (see Table 1 and Refs. [8–15]).

Further on, we make use of the values $E_a^T = 0.16$ eV, assume that $\frac{S_{d1}^*}{S_{d2}^*} = 0.3$ and $N_{d0} = 10^{17}$ cm^{-3} and replace the integral in Eq. (7) with its approximate equivalent $\tau_0 \beta^{-1} \left[\frac{kT^2}{E^T + 1.85kT} \right] \exp\left(-\frac{E_a^T}{kT} \right)$ (with $\tau_0 \beta^{-1} = 5 \times 10^4$ K^{-1} and $T_0 \to 0$). Then the dependence $\alpha^{PE}(T, h\nu_{3max})$ can be calculated after the equality $\alpha_0 = 0.2$ cm^{-1} is taken into consideration. This dependence (see curve 4 in Fig. 2a) fits well the experimental curve obtained for the BSO:Al crystals (curve 2 in Fig. 2a).

It is noteworthy that the optical (E_a^O) and thermal (E_a^T) activation energies are essentially different for the same local levels inside the forbidden gap (see Table 1). The ratio $E_a^O / E_a^T = f$ lies in the range 2–10, which indicates a strong electron–phonon interaction.

The 'reverse' dependences $\alpha^{PE}(T, h\nu_{i max})$ for the BSO, BSO:Al and BSO:Ga crystals are characterized by small α^{PE} values, which depend weakly on temperature. This fact confirms depletion of the donor levels under study. In the case of BSO:Cr crystals, the 'backward motion'

of the $\alpha^{PE}(T)$ dependence is more complicated. This can be due to temperature dependence of the intra-centre absorption in the absence of PCE (see Fig. 3b).

In the case of BSO:Cr crystals, one has to take into account that the Cr^{2+} ions occupy the positions of Bi^{3+} and can also be acceptors. Here the PCE is due to changing charge state of chromium according to the scheme $Cr^{2+} - e \rightarrow Cr^{3+}$ [6]. Apparently, the $\alpha^{PE}(T, hv_{i\max})$ curves superpose the two types of behaviour: a stepwise decreasing curve due to thermal filling of the acceptors (with the conversion $Cr^{3+} + e \rightarrow Cr^{2+}$) and a U-shaped curve characterizing the temperature dependence of the intensity of intra-centre electronic transitions. The first type prevails at the stage when the crystal is heated and the PCE erased. The second type characterizes the 'reverse' temperature dependence $\alpha^{PE}(T, hv_{i\max})$ (with $hv_{5\max}$) which occurs when the temperature is lowered in the absence of PCE (see Fig. 3b).

4. Conclusion

Summing up, we have shown that the joint studies of the spectra of impurity-associated optical absorption in a photo-excited state and the temperature dependences of photo-induced absorption enable one to find the optical- and thermal-activation energies for the same local levels in the forbidden gap. We have demonstrated this general result using the pure and doped BSO crystals as specific examples. Our approach also enables determining the strength of electron–phonon interaction.

In order to determine the thermal activation energy for the optically active centres, which are responsible for the PCE, the methods adopted in the thermally activated current spectroscopy have been applied to the temperature dependences of the photo-induced optical absorption.

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Анотація. *Запропоновано метод термічно активованої спектроскопії в застосуванні до оптичного поглинання домішок. Метод полягає у вимірюванні температурної залежності оптичного поглинання в широкозонному напівпровідниковому кристалі та аналізі температурної похідної цієї залежності. Згадана методика дає змогу визначити енергії термічної та оптичної активації та силу електрон-фононної взаємодії для домішкових центрів.*